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ISHMAEL: In-situ Sample Handling Modular Analytical Experimental Laboratory [1]

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Abstract—In-Situ instruments are an integral part of mission designs for exploration of planetary surfaces. A technology gap exists today between sample acquisition and sample analysis tools. Integrated science payload packages need an integrated sample handling system.

We are developing a set of modular, reconfigurable, rapid prototyping components for sample manipulation. One approach is to transport and manipulate samples from a few microns up to ~100 microns in diameter by a carrier fluid contained in microfluidic manifold of channels.

Our tinker toy set will consist of passive and active components. The passive components are easily stackable in three dimensions and can be built into complex distribution geometries to service many instruments. The active components will allow for sample sorting, gating and immobilization. We will use dielectrophoretic technology to manipulate particles in liquid flow.

Certain science tasks can be performed by the sample handling system itself, such as dielectric spectroscopy or microscopic analysis of caged particles.

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1. INTRODUCTION

In-Situ instruments are an integral part of mission designs for exploration of planetary surfaces. Since remote sensing instruments cannot answer many of the science questions now being posed about planetary surfaces. Analysis of surface samples is required to examine issues of detailed mineralogy and structure, biosignatures, and weathering. In turn, this scientific program demands a landed instrument that can acquire, manipulate and analyze samples with a suite of *in-situ* technologies.

Figure 1. A technology gap exists today between sample acquisition and sample analysis tools. Integrated science payload packages need an

integrated sample handling system to supply the many instruments with samples.

In the conduct of an *in-situ* program, instruments are deployed to remotely select interesting sites, rocks, strata, etc., and rover navigation is directed to these targeted samples for acquisition and analysis. Samples may originate from the surface, subject to weathering and processing, or, more generally, fresh samples will be obtained from below the surface or within a rock sample by scoops, chippers, drills or corers; several approaches are in development for this aspect of sample acquisition. The final step of placing the prepared sample into an instrument yet remains to be done, and a wide array of sample handling methods has been proposed in the past, each tied to a specific science instrument and environment.

As designs for *in-situ* missions become more sophisticated, ever more substantial mass and cost investments in instrument-unique sample handling infrastructure are required. We are investigating technologies to bridge the gap between the sample and the instruments, as shown in Figure 1, and reporting on some preliminary experiments.

We intend to eventually develop a set of *in-situ* instrument sample handling building block components that can be easily reconfigured for specific instrument arrays and requirements. These components will transport and *manipulate* samples from a few microns up to ~100 microns in diameter by a carrier gas or fluid contained in a microfluidic manifold of channels.

The building blocks will consist of valves, sample channel interconnects, inlet ports, interlocks to instruments, pumps, sample splitters, active gates and sorters, bi-directional channels and areas that can be optically interrogated. The technology is easily stackable in three dimensions and can be built into complex distribution geometries to service many instruments. Particulate samples are moved around the manifold.

For a single instrument, a dedicated sample scoop on an arm may be all that is required. For example, the Mars Organic Detector (MOD), selected for the Mars '05 mission as one of the HEDS payloads, requires a relatively large sample of ~3 cm^3 which is then effectively destroyed during analysis. The plan is to integrate the instrument with a scoop that can fill the MOD sample chamber as needed. However, other science instruments or integrated packages may analyze particulate samples from surface dust, filtered particles from aeolian processes, or products from subsurface drilling. Missions to icy bodies or the Martian polar caps could require sample handling of embedded particulates or ice grains (not necessarily water).

For integrated science programs with many instruments, each of which needs to be supplied with samples, a sample handling and distribution technology is essential. A single sample handling and distribution system decreases the scope and complexity of the individual instruments as they no longer need to manipulate their own samples individually, and reductions of mass and power can be realized. The best science may come from examining the *same* sample with different instruments, so a sample system may be required to isolate and move specific samples. Another desired feature is the distribution of aliquots of the same sample to multiple instruments. A single system also reduces mission risk. Although it is single point failure site, a single system can be designed and tested more extensively than *n* different individual systems of each instrument, each with different design and testing program.

What sort of missions and science would such a sample handling system support? There are several broad classes of missions and instrument packages that will require complex sample systems.

2. Science Needs

The proposed sample handling and distribution system will meet the needs of science instruments that analyze particulates, whether they are mineral or ice, as well as liquid samples by default. Planetary scientists are interested in a wide range of information about particulates, including size distribution, chemical and isotopic composition, mineralogy, and presence of organics and volatile fractions, if any. Particulate sampling is crucial for examining fluvial deposits, aeolian or aerosol samples and stratigraphy from sub-surface drilling. To obtain the required information, a science payload of many measurement instruments and techniques can confidently be expected, e.g. mass spectrometers, microscopes, Raman spectrometers, etc.

Our technology will provide a sample stream that can be presorted and gated to specific instruments. By design it is a reconfigurable sample handling system, constructed of elemental building blocks that can be tailored for specific missions and instrument complements without a costly redesign.

Mars

Polar Layered Deposits (PLD) on Martian polar caps are considered to be the depository of the planet's geological and climate history [1,2]. The deposits are believed to be ~10^5-10^8 years old and consist of aeolian dust, volcanic ash, water and carbon dioxide ice. They may contain the planetary record of obliquity cycles, impact events, volcanoes, seasonal dust storms, supernova events and other global processes. Detailed studies of physics and chemistry of the PLD would arguably open a new page in human knowledge about Mars. Several missions are currently in different stages of planning to address specific science issues with new instrument technologies.

The Mars Climate History Mission concept [3] proposes to drill through the northern PLD down to a depth of about one-kilometer. The samples in the form of 100 micron-sized particles would be extracted from different depths and delivered to the lander via a capillary tether containing liquid krypton. The lander instrument suite on an actual mission would be designed to perform multiple analyses to address science objectives of the mission. The Mars Climate History mission study team developed a scheme for a sample handling system that would interface with an extensive instrument set called for by the mission science objectives. Figure 2 sketches the complexity that a sample distribution system can take for such an integrated science package.

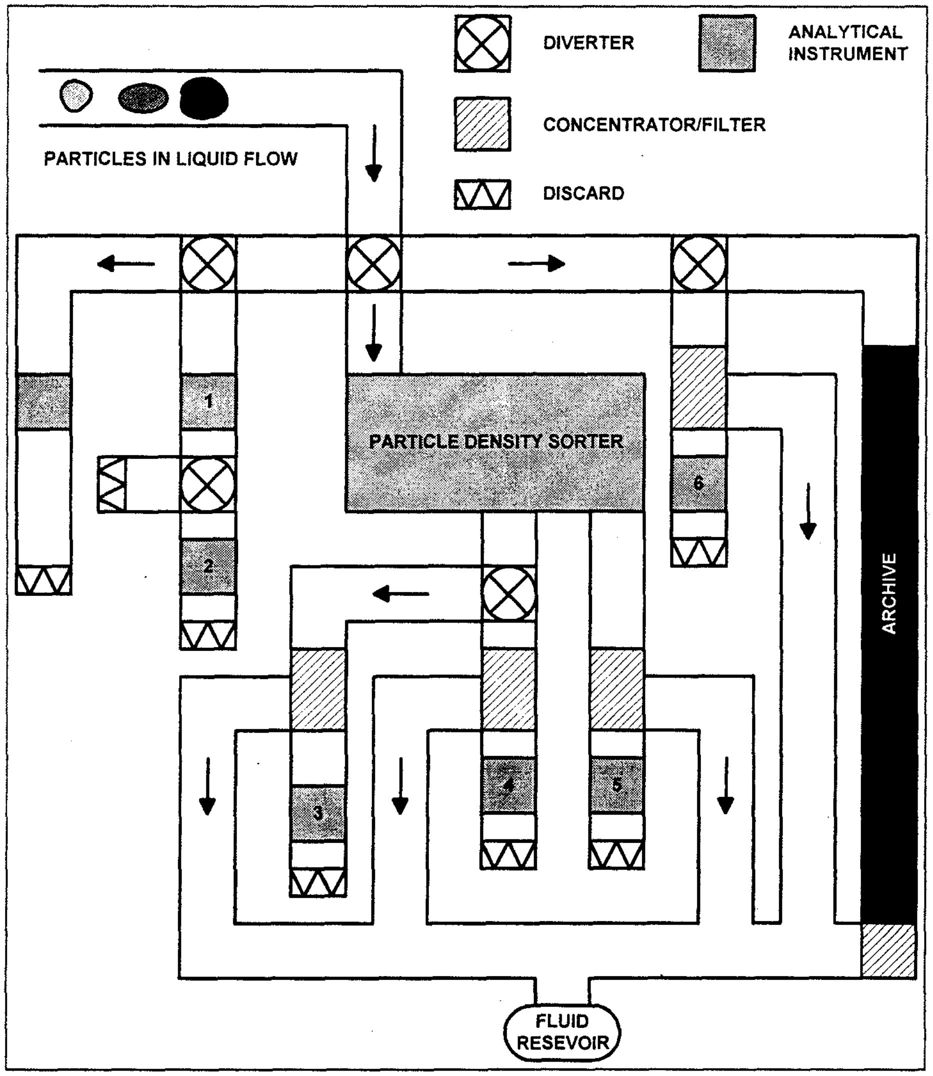


Figure 2. Sample handling scheme proposed for Mars Climate History Mission concept study. Composite ice/dust samples are split among various instruments, which accept particles from the fluid stream. Instruments are designated as follows: 1 – Deconvolutional Confocal Microscope, 2 – Raman Spectrometer, 3 – Electron Paramagnetic Resonance Spectrometer, 4 – Mass Spectrometer for ice analysis, 5 – Mossbauer spectrometer, 6 – Mass Spectrometer for composite particles analysis.

This mission concept study revealed that integrated sample handling methodology is a key enabling technology needing further development for the mission. Participants of the Mars Geo-chronology Workshop (June 4-6, University of Illinois Chicago) have also noted the importance of sample handling and delivery to instruments [2].

Martian surface science involves the analysis of fluvial and aeolian deposits, along with aerosols to understand deposition and layering. Analysis of these features requires a variety of diverse measurements on samples--composition, mineralogy, particle size distribution, organic components and volatiles. In each case, the natural sample is particulate in nature and a science package would contain many instruments.

Europa

The possibility of a liquid ocean under the ice crust on Europa [4] has in the recent years attracted a great interest in this planetary body as a possible harbor for extraterrestrial life. A Europa Orbiter Mission is planned for launch in November 2006, probably to be followed by a Europa landed mission. Analogous to the Mars Climate History Mission, the goal of the *in-situ* exploration on Europa would be to penetrate through the ice cover (possibly acquiring samples on the way) and explore the ocean underneath. In this case, there is no need for a carrier fluid in the sample handling system, as the sample itself is a fluid. Since the passive and active elements of the proposed sample handling system are basically plumbing, they are well suited to aqueous samples. Particulates in the fluid can be moved and sampled as before. In this case, any measurement ability of the sample handling system itself becomes an important addition to mission science.

Mission scenarios show that there are really two problems to be solved—sample transport and sample distribution and manipulation. Subsurface explorers, for example, acquire the sample remotely as they bore through the surface. Samples need to be transported back to the analysis lab on the lander. It is not possible for the explorer to repetitively return to the lander with samples, especially if stratigraphy is a major science goal. Once samples are at the instrument package, it now becomes necessary to distribute them. A particular mission may need either capability or both, depending on the instrument suite and science goals.

Instrument Specific

With the growing recognition of the feasibility and importance of *in-situ* planetary missions, a wide variety of analytical instruments are being developed for planetary exploration. Not all lab workhorses can be miniaturized into field ponies; however, there is still an amazing variety of instruments already flight qualified or nearing flight readiness. Rather than compiling an exhaustive listing of such projects, Table 1 shows a selection of instruments that any *in-situ* package would draw from.

	localized sample	analysis pressure	particulate samples?
mass spectroscopy	no	low	yes
optical microscope	yes	any	yes
electron paramagnetic resonance	no	any	yes
Raman spectrometer	no	any	yes
atomic force microscopy	yes	any	yes
scanning electron microscopy	yes	any	yes
laser induced breakdown spectroscopy	yes	any	yes
luminescence dater for alluvial deposits	no	any	yes
NMR	no	any	yes
Mossbauer spectrometer	no	any	needs milligrams
evolved gas analyzer	no	low	yes
capillary electrophoresis	yes	any	yes

Table 1. Tools of the trade. Representative *in-situ* instruments.

3. SAMPLE HANDLING REQUIREMENTS.

Why microfluidics?

For *in-situ* missions, it is beneficial to separate the tools for *sample acquisition* (rovers, penetrators, balloons etc. which are the platforms for the drills, corers, etc.) and *sample analysis* (analytical instruments). The former are usually mobile, resource hungry, small and are a severe vibration and shock environment making it difficult to put science instruments on-board. The latter are often stationary (lander-based) and off-line, meaning that the analysis time is frequently longer than the sample acquisition time. This general structure dictates a need for an interface between the acquisition and analysis systems, as well as a sample distribution manifold within the instrument suite.

We have chosen a technology that is well suited to sample transport, namely fluidics. This technology easily lends itself to movement of entrained particles and fluid samples. In the macro world, there are plenty of examples to serve as models such as coal slurry piping. More importantly, there is a significant technology base from laboratory automation and biochips in the area of microfluidics that can be used for development. Since we are looking at significant fluid and particle samples, perhaps a better term would be "millifluidics", as we are not interested in nanoliters, the typical scale that biochips strive for. We have chosen a technology that can be scaled up to provide channels and active elements on the scale required for *in-situ* instruments.

A sample handling system can be expected to perform a variety of functions to meet the science instrument needs. In general, without reference to specific missions or instruments, we think that a sample handling system should be able to do the following:

- Provide passive and active sample splitters -to distribute samples among a manifold
- Take in samples through an inlet at high pressure to interface to a low-pressure instrument
- Provide valving
- Provide pumps for the working fluid and sample
- Provide inlet and outlet ports to interface to instruments
- Be able to isolate and move specific samples in the system
- Unclog the sample system
- Be easily cleaned for contamination control, especially for biodetection instrument packages
- Isolate a fluid sample aliquot

Each of these needs can be mapped into a candidate instrument or science experiment for Mars or Europa. For example, a Mars Polar lander that bored through the ice cap and transported samples to an instrument suite would require a high-pressure sample inlet. Another such example would be a Europa Ocean explorer, which would be working against the pressure overburden of ice and liquid when exploring the subsurface ocean.

A suite of instruments can be expected to use a screening technology to identify sample targets of particular interest. In that case, instrument A identifies a sample as being suitable to examination by instrument B and that particular sample needs to be isolated and delivered to B.

Missions that use this sample manifold may have instruments that work at quite different pressures than sample acquisition. We will develop, optimize and characterize the system at a fixed pressure and provide a step-down regulator to match high-pressure sampling to the lower fixed system operating pressure. Besides making the system more robust, this approach means that reconfiguration of the system for various missions is minimized as a new set of operational parameters is not needed.

4. TECHNOLOGY SUBSYSTEMS

Particle Transport. Clog and Play.

We have performed particle transport experiments with several meters of thick walled capillary, as has been proposed for the Mars Climate History Mission with a subsurface explorer. Samples are transported to the lander for analysis in a capillary tether between the explorer and the lander. The capillary is a flexible plastic with 100 μ walls and a 100 μ clear channel. One kilometer of the capillary will fit a spindle about 3 " in diameter.

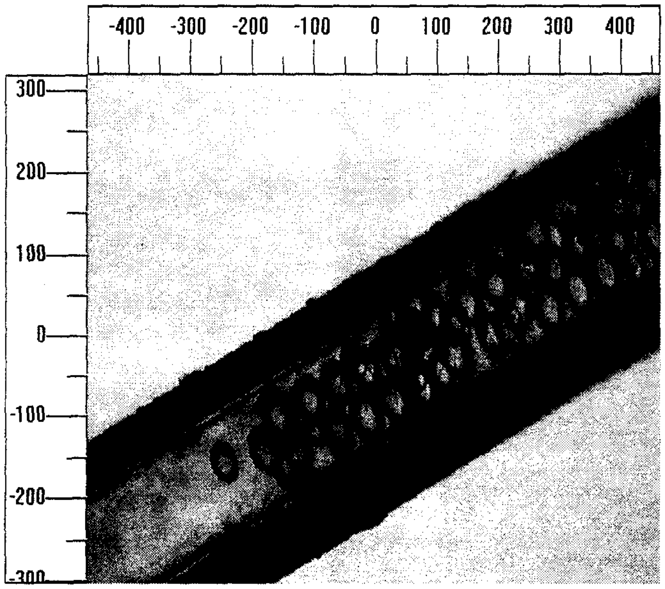
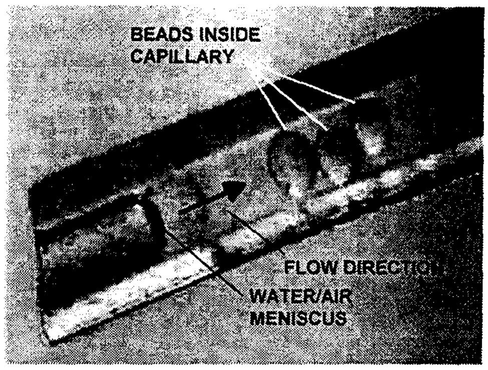
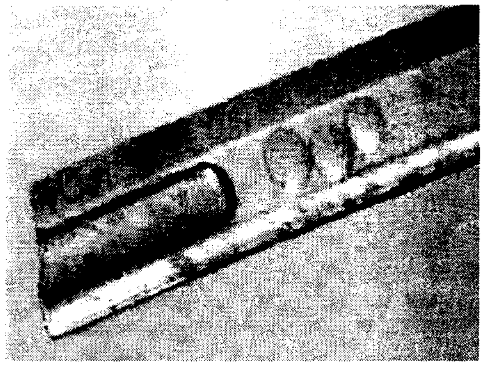


Figure 4. Glass beads form a closely packed clog inside a 100 μm ID capillary. Units on the rulers are microns.

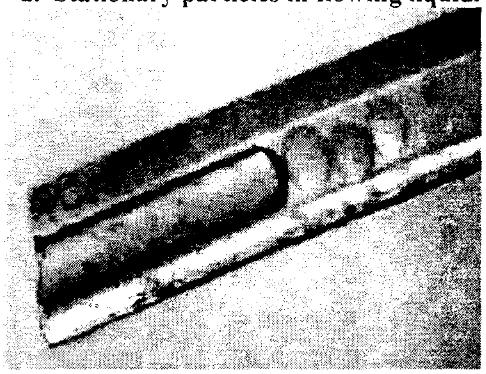
An obvious problem in particle transport through a manifold is clogging. Our initial experiments show that what is true in the macro world stays true in millifluidics – *it is easy to clog the pipe.* Figure 4 shows a hard packed clog created by 10-30 μm glass beads during an attempted transport through a 100 μm ID capillary. The beads were carried in water while a syringe pump provided pressure. Experiments with larger particles greater than half the internal diameter show that clogs do not form in this case as there is no interparticle contact.



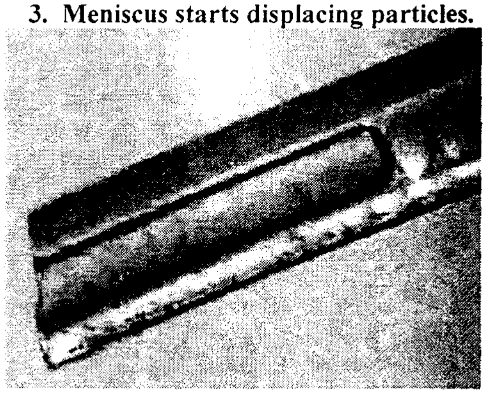
1. Initial State.



2. Stationary particles in flowing liquid.



3. Meniscus starts displacing particles.



4. Displacement continued.

Figure 5. 80-100 μm size glass beads inside 100 μm capillary. Particles adhere to the surface but move with meniscus at the air-water interface.

We have also done experiments transporting Martian Simulant and see similar clogging results. It appears that the clogs form in small pieces that travel together and accumulate at sites to make much larger clogs. By reversing fluid flow, we can usually breakup the clog. Similarly, clog breakup happens piecemeal, with the clog dispersing in sections, rather than the complete breakup into individual particles.

We have discovered that the beads do not necessarily follow the liquid flow; instead they can follow the moving air/water meniscus. As surface tension forces are very strong on the microscale, it is efficient for particle transport, as demonstrated in Figure 5.

Four different frames show particles inside a capillary. The arrow indicates flow direction. Liquid flow itself does not displace particles, as is observed in first two frames. However, when the meniscus reaches the beads, they begin moving together with meniscus, see last two frames as well as possible approaches for clog removal. While the dominant force here is most likely surface tension, there may be electrostatic forces as well, since it is easy to charge these particles. This suggests that we can use bubbles as a tool to separate and manipulate particles.

Particle agglomeration and sticking is a problem not only for this approach to sample transport and handling. Particles will stick, most likely due to electrostatic forces, to walls and any surface involved in the sample chain, whether robotics, conveyor belts, surface acoustic waves and so on are used to move samples. Aside from problems related to sample motion, this poses an issue for sample contamination as succeeding samples may contain particulates from previous samples that adhered to the wall and became unstuck later.

We conclude that there are two related approaches to controlling clogs in the transport system. One is to control the ratio of the particle size to the fluid flow channel, perhaps by prefiltering. A second is to space the particles along the transport channel. For a subsurface explorer, this is done by spacing the sample acquisition in time.

Passive components. Modular manifold.

Nanostream, a Pasadena microfluidics company, can provide a base for rapid prototyping of passive elements with its new reconfigurable and 3D stackable microfluidics technology [5]. The technology supplied by Nanostream provides a number of advantages for this effort. Fluid mechanic calculations do not apply due to the small Reynolds number so the typical approach to microfluidic systems is trial and error fabrication and testing. While lithography and some other fabrication techniques are cost effective at high volume (>1 million units), prototyping can be very costly for system development. In addition, prototyping with such methods as slow and can take 4-12 weeks per cycle.

Nanostream's polymer based fabrication technique is inexpensive and timely, taking as little as a few hours to design, make and test a component. This ability will permit us to build and test a wide variety of components and assembly them into systems. Much of Nanostream's contribution will be our passive components, although the valving and pumps are, of course, active elements. The Nanostream fabrication technology results in a monolithic block containing the entire microfluidic system for a compact and robust system.

As an example, Figure 3 shows some basic sample handling modules fabricated by Nanostream. Note that these are designed solely for liquid samples—one of the challenges is to reconfigure the dimensions and devices to function with particulates.

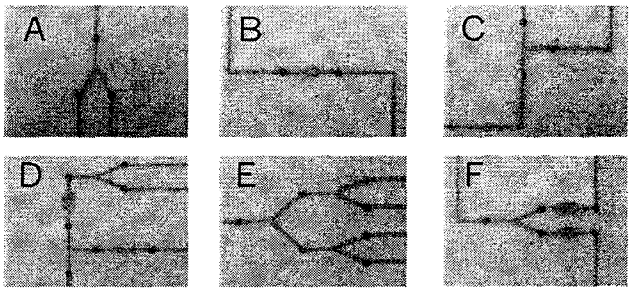


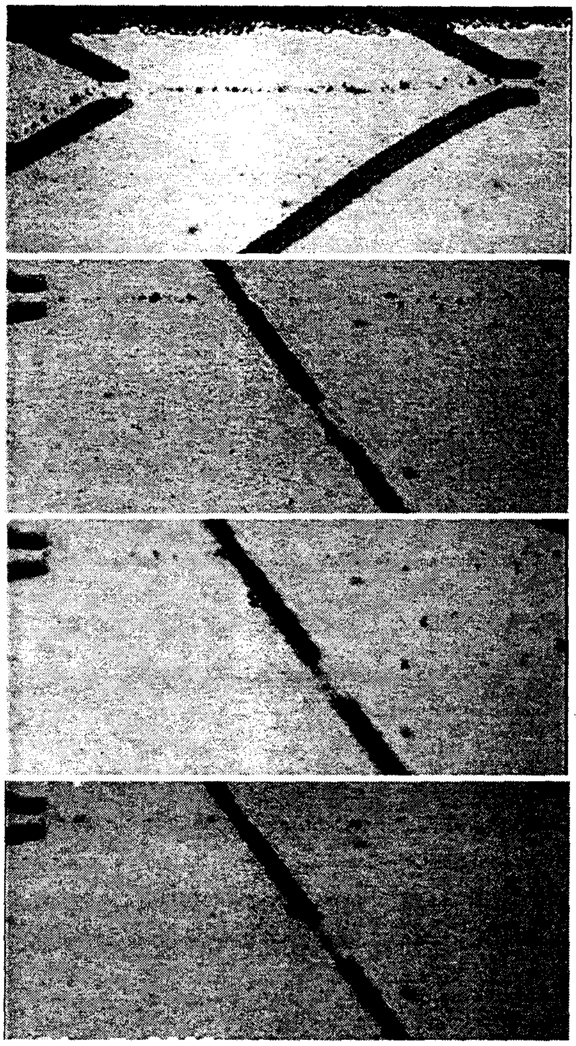
Figure 3: Various NanoModules™ are constructed that perform basic functions such as splitting (A), filtering (B) and valving (C). These modules can be combined in various configurations (D-F) to perform more complex functions. The channels in these devices are ~1 mm wide by 40 μm tall. Images courtesy of Nanostream.

Active Particle Manipulation. Dielectrophoretics.

For active sample manipulation and sorting, we are using dielectrophoretic technology, which also comes from the biochip world [6,7]. Developed to sort, gate and trap biological cells, the same technology is applicable to mineral particulate or ice samples in a fluid stream. The technology, originally developed in Prof. Fuhr's labs in Humboldt University (Berlin, Germany) is being commercialized today by a German startup Evotec [8].

Dielectrophoresis uses high frequency (1-20 MHz) RF to create an induced polarization [dipole] in particles which then move when exposed to an inhomogeneous electromagnetic field. It has been used to manipulate or move bubbles or cells [9,10] and to move fluids. The current use of the technology is for microfluidic "lab-on-a-chip" to perform cell manipulation and sample preparation for gene chips and other types of parallel immunoassays. Evotec has demonstrated sorting, transport and manipulation of cells, latex beads and resin beads with this method.

One feature of dielectrophoretic manipulation of cells is that it can be rapidly turned on and off based upon some other criteria. For example, one can gate particles into one side of a sample splitter or another by establishing a repelling field gradient at the entrance to one channel and an attractive one at the other. The next particle, which may need to go oppositely, can be forced to do so by reversing polarity.



A
B
C
D

Figure 6. Mars soil analog particles (JSC-1) in the dielectrophoretics chip. Particles are below 40 microns size are embedded in water flow. The flow direction is from left right. Dark strips are electrodes to which RF electric field is applied. Electrode cross-section is 20 microns. Electrode thickness is much smaller than the depth of the channel, 40 micrometers. Thus, electrodes do not form a physical barrier for liquid and particles. When field is applied to electrodes, particles are repelled from them and particle motion is controlled by the balance of hydrodynamic and dielectrophoretic forces. (a) Particles aligned by two electrodes in the "funnel" arrangement. (b), (c) and (d) Three frames showing stream of aligned particles as being size sorted at an electrode. Larger particles track electrode while smaller ones follow the flow. To the right from the electrode the particles continue their laminar flow.

Since the forces are a result of the applied RF, this technique will work for mineral and ice particulates as well as cells—the major criteria is that the complex permittivity of the sample be different than that of the carrier fluid.

While there is a lower limit of ~ 35 nm for dielectrophoretic movement of particles, the limits on upper size are practical--power and thermal concerns dominate. The lower limit arises from the fact that the dielectrophoretic forces are proportional to volume, why competing hydrodynamic forces from the carrier fluid are proportional to the area. As the particles become smaller the dielectrophoretic forces decrease and eventually are not sufficient to control the particle. Movement of 100 μm particles is not an issue, as we have demonstrated.

Active manipulation of particles with dielectrophoretic forces can also create a trap or cage using appropriately phased frequencies on appropriate electrodes [6,11]. These cages are ideal for interrogation or manipulation of individual particles. They can be optically or spectroscopically analyzed and then ejected through a port for further analysis by one of the science instruments.

The sample handling system, could, under some circumstances, also be a science instrument. For example, dielectrophoretic systems with special geometry manifolds can provide particle sizing. Dielectric spectroscopy performed on single particles on the manifold can measure electrical properties such as complex conductivity and permitivity [12,13].

We have conducted initial tests of soil particles manipulation by Evotec's dielectrophoretic system with water as a carrier fluid. Some results are presented in Figure 6. In this case, we are looking down on the top of the fluid flow channel, which is about 40 μ deep and about 1 mm wide. The large width we can use with this approach significantly reduces the possibility of clogging.

Figure 6 shows frames from a movie using dielectrophoretics to sort Martian Soil Simulant by size. Only particles smaller than 40 microns are let into the system. Particles enter with the flow from the left and are aligned into a single stream with the V-shaped electrodes, Figure 6a.

Since the fluid flow is laminar, once the particles are located at a certain height in the stream, they stay in it. In the second and third frame, the particles are being sorted by size with a straight and curved electrode. The larger particles travel further down the electrode and are then swept into the stream flow when the hydrodynamic force equal the dielectrophoretic force. In this manner we can create a laminar flow of particles in which the size increases towards one side of the channel.

Another idea for such "Science in the plumbing" approach is to build parts of the manifold from specific materials. Particle interactions with these "patch pipes" will tell us about particle properties similarly to the approach of patch plates implemented on Mars Environmental Compatibility Assessment (MECA) payload [14], originally designed for '01 Lander. On MECA, 72 patch plates were to be exposed to Martian particles to study a variety of dust physico-chemical properties. In our case the test materials integrated in the manifold can be hydrophilic/hydrophobic (to classify particles on their surface adhesion properties) or magnetic (to segregate and accumulate magnetic particle fraction).

5. CONCLUSIONS.

The goal of our investigation is to come up with a set of tools and recipes for transport and manipulation of solid particles. We believe that the majority of the tasks can be accomplished by embedding particles in a flow of chemically inert liquid. The particles then are carried along the passive manifold and are actively controlled by dielectrophoretic elements.

6. ACKNOWLEDGEMENTS.

The research described in this publication was carried out at the Jet Propulsion Laboratory, California Institute of Technology, under a contract with the National Aeronautics and Space Administration.

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